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ARTICLES ON SOVIET POWDER METALLURGY

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## ARTICLES ON SOVIET POWDER METALLURGY

[Following is a translation of several articles on powder metallurgy published in Doklady Akademii Nauk SSSR (Reports of the Academy of Sciences USSR), Izvestiya Akademii Nauk SSSR (News of the Academy of Sciences USSR), and Izvestiya Vysshikh Uchebnykh Zavedeniy, Tsvetnaya Metallurgiya, Chernaya Metallurgiya (News of Higher Educational Institutions, Non-Ferrous Metallurgy and Ferrous Metallurgy series) and Vestnik Mashinostroyeniya (Herald of Machine Building). Bibliographic information is given with individual articles.]

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## SOME ASPECTS OF THE HYDROSTATIC PRESSING OF POWDERS

M. Yu. Bal'shin and A. P. Dubrovskiy

Presented by Academician Bozhvar 3 August 1960

Moscow, *Doklady Akademii Nauk SSSR* (Reports of the Academy of Sciences, USSR), Vol 136, No 2, II January 1961, pp 332-335.

Hydrostatic (isostatic) pressing of powders [1] has so far been used only up to pressures of 10-12 kg/mm<sup>2</sup>, which gives no proper basis for formulating the laws governing the behavior of powders in response to such pressing.

We have used pressures of 10-60 kg/mm<sup>2</sup> with powders of electrolytic copper (poured density 1.82 g/cm<sup>3</sup>, tamped density 2.86 g/cm<sup>3</sup>), vortex iron (1.72 and 2.56 g/cm<sup>3</sup>), electrolytic nickel (2.57 and 3.70 g/cm<sup>3</sup>), and reduced molybdenum (tamped density 3.22 g/cm<sup>3</sup>). The particle sizes were less than 10  $\mu$ , except for the iron (particles < 90  $\mu$ ). The powders were pressed into rods within elastic sheaths (initial diameter 11 mm, length 65 mm; linear dimensions reduced by 25-30% by pressing). The specimens for use in determining the strength in compression were cut into cylinders having a ratio of height h to diameter D of about 0.7; preliminary tests on specimens cut in various orientations from large pressings showed that the strength in compression was the same in all directions. The pressure was applied perpendicular to the ends in the tests.

Conventional pressing in steel dies was used at the same pressures on the same powders for comparison. Specimens with h/D  $\approx$  0.7 were used to measure the density as a function of pressure for both methods of pressing, the density being measured by weighing in air and in a liquid.

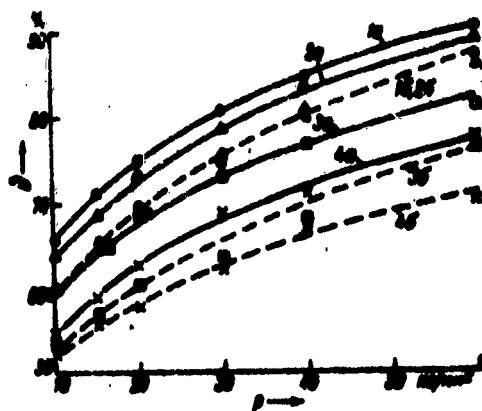


Fig. 1. Relation of  $\theta$  to  $p$  for a) hydrostatic pressing and b) ordinary pressing; 1) Cu; 2) Fe; 3) Ni; 4) Mo.

tion being some 20-30% for fine copper powder. The smallest effect occurs with the coarse iron powder (reduction of only 10-20%).

Figure 2 shows  $\log \theta$  in relation to  $\log p$  for both forms of pressing; the relation is linear, so [2] the relation of  $\theta$  to  $p$  may be put as

$$\frac{p}{p_{\max}} = \theta^m, \quad (1)$$

in which  $p_{\max}$  is the maximum pressure (that giving  $\theta = 1$ ) and  $m$  is a constant. That is, the resistance to deformation is specified by  $p_{\max}$  and  $m$  (see Fig. 2 for values of  $m$ ), which are larger for hydrostatic pressing, except in the case of Mo. Table 1 gives  $p_{\max}$  together with the hardness of the metals; the  $p_{\max}$  for the softer metals (Cu and Fe) are almost the same for both methods, but those for the harder metals are less when hydrostatic pressing is used (by 24% for Ni and by 32% for Mo).

Table 2 gives  $\sigma$  (the strength in compression) for Cu (a typical soft metal) and Mo (a typical hard one). Ordinary pressings gives considerable anisotropy in ; the ratio of  $\sigma_1$  to  $\sigma_2$  is particularly large at low pressures, and it is larger

The conventionally pressed specimens were tested in compression along the pressing axis (cylinders 15 mm in diameter having  $L/D \approx 0.7$ ) and at right angles to that axis (rectangular blocks of side  $a$  perpendicular to the pressure of 6 mm).

Figure 1 shows the relative density  $\theta$  as a function of pressure  $p$  for both forms of pressing; hydrostatic pressing is clearly always the more effective. The reduction in the pressure needed is especially pronounced for fine and hard materials (nickel and molybdenum; hydrostatic pressures need be 30-50% less to give the same density), the reduc-



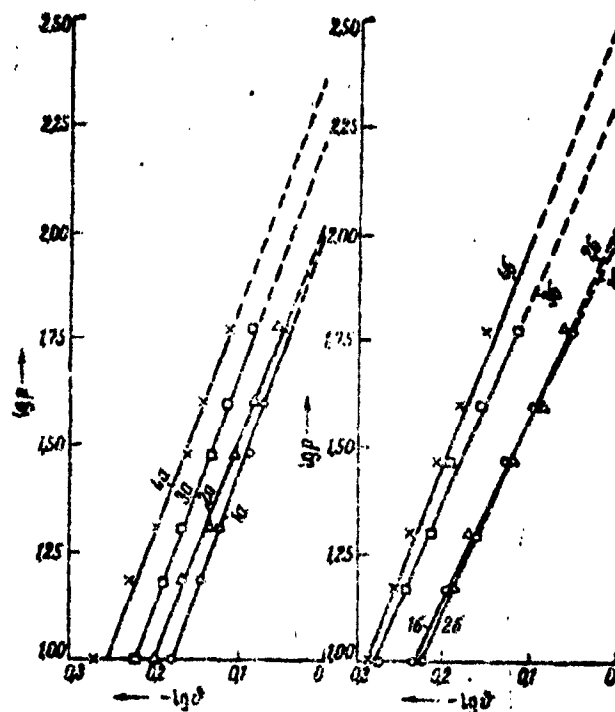


Fig. 2. Relation of  $\log p$  to  $\log \theta$  for a) hydrostatic and b) ordinary pressing: 1) Cu; 2) Fe; 3) Ni; 4) Mo;  $m$ : 1a) 5.65; 2a) 5.26; 3a) 5.5; 4a) 5.4; 1b) 4.45; 2b) 4.66; 3b) 4.82; 4b) 5.33.

for Mo (hard) than for Cu (soft). A hydrostatically pressed material has the same strength in all directions, and this is roughly equal to the highest strength in ordinary pressing (for Cu) or rather larger than it (by 20-130% for Mo).

Table 1

Metal	$H_B$ , kg/mm <sup>2</sup>		$P_{max}$ , kg/mm <sup>2</sup>	
	Ann.	W. h.	Hydro	Ord.
Cu	40	to 120	104	108
Fe	60	" 180	111	112
Ni	90	" 250	171	224
Mo	150	" 270	244	360

In ordinary pressing, the pressure  $p$  is reduced by an amount  $\Delta p$  as a result of friction at the walls of the die [2]:

$$\frac{\Delta p}{p} = \mu \frac{h}{D}$$

(3)

in which  $\xi$  ( $\approx 0.3-0.4$ ) is the lateral pressure coefficient,  $h/D$  is here  $\approx 0.7$ , and  $\mu$  (the coefficient of friction) is 0.15 to 0.35; then  $\Delta p/p$  is 0.1 to 0.4 in our case, so the effective pressure is 10-40% less than the nominal pressure. This shows why  $p_{\max}$  for hydrostatic pressing is less than that for ordinary

Table 2

Material	Pressure, kg/mm <sup>2</sup>	$\sigma$ for ord. pressing, kg/mm <sup>2</sup>		$\sigma_{  }/\sigma_{\perp}$	$\sigma$ for hydro., kg/mm <sup>2</sup>
			$\perp$		
Cu	10	4.68	0.74	6.3	4.26
	20	9.58	3.18	3.0	8.24
	40	19.4	10.1	1.9	15.4
	60	22.4	13.8	1.6	22.9
Mo	10	0.28	-	-	0.33
	20	0.84	-	-	1.94
	40	4.35	-	-	7.5
	60	12.2	1.0	12.2	18.1

pressing (by 1% for Fe, by 32% for Mo). Table 1 shows that  $p_{\max}$  is determined mainly by the hardness (internal strength) of the particles, being between the hardness values for the annealed and maximally work-hardened states in most cases. Only in the case of molybdenum (the hardest metal) is  $p_{\max}$  for ordinary pressing substantially greater than the hardness in the maximally work-hardened state; this is to be attributed to frictional losses. The difference between the  $p$  for a given degree of compression also corresponds with the  $p_{\max}$  and  $n$  for hydrostatic and ordinary pressing.

There are two explanations for the differences between the hard and soft metals.

1. The friction and frictional losses in the dies are much larger for the hard metals, in which case the differences in the  $p_{\max}$  and  $p$  would be larger for the harder metals. This was tested by reference to the ejection pressure for  $h/D = 0.7$  for these powders (the pressure is almost equal to the pressure loss at the wall). In every case the ejection pressure was about 40% of the total pressure (38% for Mo, 41% for Cu), so this

possibility is eliminated.

2. Seizure of the particles of the softer metals <sup>13</sup> appears more probable, for this greatly increases the adhesion between particles and hence the internal friction (i.e., the resistance to deformation). The effect is especially prominent in the hydrostatic pressing of soft metals, for it operates in all directions here, whereas in ordinary pressing it operates in one direction only. That is,  $P_{max}$  differs little between hydrostatic and ordinary pressing for soft metals (the absence of external friction is balanced by the higher internal friction) but differs greatly for the hard metals.

That is, hydrostatic pressing is especially effective for forming hard-metal powders to high density and strength.

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CSO: 1879-N

A RADIATION METHOD OF PREPARING FINELY DIVIDED  
METALS AND OXIDES

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Moscow, *Izvestiya Akademii Nauk SSSR* ( *News of the Academy of  
Sciences, USSR* ), Vol No 9, Sep 1962, pp 1,690-1,692.

Some problems in the use of active solids involve a decision on the conditions of preparation that give the maximum specific surface. The last stage of preparation is often a topochemical decomposition of some compound (salt or hydroxide, say) or a reduction; but such processes require high temperatures, at which sintering accompanies the formation of the new phase. This often causes difficulty in the preparation, especially for oxides.

Radiation-induced decomposition of a solid compound occurs at much lower temperatures  $\Delta T$  and so may not be associated with appreciable sintering. This makes it of interest to examine the radiolysis of salts and hydroxides for the preparation of metals and oxides as powders.

We have used the hydroxides of Zr, Fe, and Al, as well as nickel oxalate, copper oxalate, and basic copper carbonate; these were made in solution from the nitrates with ammonia (hydroxides) or oxalic acid (oxalates), apart from the copper carbonate, which was the commercial chemically pure grade. The source was the electron beam from the linear accelerator at the Institute of Physical Chemistry, Academy of Sciences of the USSR. The specimen (1-2 g) was placed in an aluminium cell fitted with an external cooling jacket; this cell was closed with an aluminium foil (10 mg/cm<sup>2</sup>). The thickness of the layer

of powder corresponded roughly to the extreme range of the electrons. The cooling jacket was fed with water for dose rates up to  $10^4$  rad/sec; liquid nitrogen was used instead for dose rates up to  $10^6$  rad/sec. These measures ensured that the temperature of the powder did not exceed  $40-50^\circ$  (as read by a thin chromel-alumel thermocouple placed in the middle of the layer). The irradiations were performed in air or with nitrogen flowing over the specimen.

The energy absorbed by the specimen was measured by placing between the window of the accelerator and the sample a monitor ionization chamber having walls of aluminium foil almost transparent to electrons [27]. The chamber was calibrated by reference to the current reaching an electrode placed at the bottom of a glass cell. The ionization current was kept constant. The dose rate was calculated from the current reaching the electrode in the glass cell (without a specimen); in some cases it was checked by means of ferrous sulfate and cerium methods.

The irradiated specimens were subjected to kinetic and X-ray analysis to determine the extent of conversion and the phase composition of the products. The specific surface of the oxide or metal was determined from the adsorption of n-pentane, benzene, or cyclohexane at  $18^\circ$ , and also with krypton at  $-196^\circ$ .

Table 1

Specific Surface of Solids Formed by Radiolysis Pyrolysis from Inorganic Compounds

Initial Compound	Radiolysis			Pyrolysis		
	Cond.	Comp.	m <sup>2</sup> /g	Cond.	Comp.	m <sup>2</sup> /g
Zr(OH) <sub>4</sub>	$6 \times 10^9$ rad in air	ZrO <sub>2</sub>	300-350	Air, 400°	ZrO <sub>2</sub>	100
Fe(OH) <sub>3</sub> *	$10^{10}$ rad in air	Fe <sub>2</sub> O <sub>3</sub>	50	Air, 450-500°	Fe <sub>2</sub> O <sub>3</sub>	10
NiC <sub>2</sub> O <sub>4</sub> ·2H <sub>2</sub> O	$10^{10}$ rad in N <sub>2</sub>	Ni 95% NiO 5%	120	Vacuum, 280°	Ni 95% NiO 5%	10
CuC <sub>2</sub> O <sub>4</sub> ·½H <sub>2</sub> O	$5 \times 10^{12}$ rad in air	Cu 98% Cu <sub>2</sub> O 2%	60	Vacuum, 280°	Cu 90% Cu <sub>2</sub> O 10%	5
CuCO <sub>3</sub> ·Cu(OH) <sub>2</sub>	$7 \times 10^9$ rad in air	CH <sub>2</sub> O	40	Air	CH <sub>2</sub> O	10

\*X-ray analysis by M. Ya. Kushnerov indicates that the composition was FeOOH after drying.

All compounds showed appreciable decomposition after doses exceeding  $10^6$  rad, decomposition being extensive for ones of  $10^9$ .

$10^{10}$  rad. Radiolysis at low temperatures gave mainly the metals from the oxalates even in air, whereas pyrolysis in air at 300-350° gives the oxides.

The specific surface of a metal or oxide made by radiolysis was usually much larger than that of one made by pyrolysis, especially for the oxalates, where the ratio of the two was 10 or more. The hydroxides of Zr and Fe gave oxides of high specific surface, but  $\text{Al}(\text{OH})_3$  gave the same specific surface as by pyrolysis at 400-450°.

#### Conclusions

1. Radiolysis of solid hydroxides, oxalates, and carbonates at low temperatures produces finely divided metals and oxides.

2. Metals and oxides made by radiolysis have specific surfaces greatly exceeding those of the same materials as made by pyrolysis.

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OSO: 1879-N

## WETTING AND IMPREGNATION OF BRIQUETTES OF TITANIUM AND ZIRCONIUM

### NITRIDES BY MOLTEN COBALT AND NICKEL

L. V. Beylina and I. P. Kislyakov

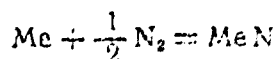
(Department of Rare-Metal Metallurgy, Krasnodar Institute of  
Nonferrous Metals)

Ordzhonikidze, *Izvestiya Vysshikh Uchebnykh Zavedeniy, Tsvetnaya Metallurgiya* (News of Higher Educational Institutions, Non-ferrous Metallurgy), No 3, 1962, pp 129-134

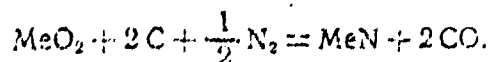
Combinations of the hard and refractory carbides, nitrides, borides, and silicides of metals of groups IV, V, and VI are of interest as new materials that are hard, heat-resistant, and wear-resistant [1-10].

Carbides of these metals are now widely used in hard alloys, but little work has been done on the nitrides [11], so studies on methods of preparation and properties are of considerable interest. In particular, this applies to wetting and impregnation by metals of the iron group and also to sintering; these topics are considered here for the powdered nitrides, nickel and iron being the metals used.

The nitrides were made from the metals:



or from the oxides:



The metals and oxides were standard commercial grades. The titanium powder was not less than 98% pure, the main impurities being hydrogen (about 0.5%), Ca ( $> 0.05\%$ ), and nitrogen ( $> 0.07\%$ ). The zirconium powder was not less than 98% pure, the main impurities being Ca, O, Si, and Ti (0.3 - 0.5% each). The  $\text{TiO}_2$  was of 99.5% purity, the impurities being oxides of iron, silicon, and sulfur (0.1 - 0.25% each) and  $\text{P}_2\text{O}_5$  ( $> 0.06\%$ ). The  $\text{ZrO}_2$  was not less than 98.85% pure, the main

impurities being  $\text{SiO}_2$  ( $\frac{1}{2}$  0.35%),  $\text{CaO}$  ( $\frac{1}{2}$  0.28%),  $\text{TiO}_2$  ( $\frac{1}{2}$  0.5%), and  $\text{Fe}_2\text{O}_3$  ( $\frac{1}{2}$  0.55%).

The nitrides were made from the metals in a resistance furnace having an alundum tube and a sillite heater; a furnace with a graphite tube as heater was used for the oxides. The nitrogen was purified by passage over heated copper turnings, then through a titanium tube, and through a column containing silicagel and phosphorus pentoxide. Table 1 gives the compositions and lattice parameters of the products (the Kjeldahl method was used for nitrogen).

Table 1

Compositions and Lattice Parameters of Nitrides

Nitride of	N, %		C, %		Parameters	
	Stoich.	Exp.**	Total	Free	Theor.	Meas.
Ti, from metal	22.65	21.6	-	-	4.230	4.2307
Ti, from oxide	"	20.2	1.17	0.22	"	4.216
Zr, from metal	13.31	12.97	-	-	4.567	4.557
Zr, from oxide	"	9.77	2.18	0.14	"	-

\*\* Error 0.1 - 0.2%

\*\* Error 0.2 - 0.5%

The metals were nitrided in two stages, the temperature being raised slowly to the maximum in each stage (1100 and 1150° respectively, first heating 3.5 hr, second 10 - 15 min., time at first maximum 1.5 hr, at second 1 hr). The oxides (mixed with lamp black) were nitrided in three stages at 1600° for times of 1 hr,  $\frac{1}{2}$  hr, and  $\frac{1}{2}$  hr, the time to reach the maximum temperature being 1 hr. The mixture was broken up and ground after each heating.

Table 2

Grain-Size Distribution (%)

Nitride of	Size, $\mu$				Mean
	-2.4	2.4-3.6	3.6-4.8	4.8-5	
Ti, from metal	31.2	33.9	22.4	12.2	2.95
Ti, from oxide	54.7	23.9	12.5	8.9	2.45
Zr, from metal	37.4	25.4	15.7	21.5	3.24
Zr, from oxide	52.1	19.6	12.9	15.4	2.47



Table 1 shows that the metals gave much better approach to the theoretical composition; the products from the oxides always contained much carbon no matter what the composition of the mixture. The proportion of carbon could be reduced only by making the content in the initial mixture equal to the stoichiometric or less; but then the product contained less nitrogen, because some oxygen remained. That is, it is not possible to make nitrides approaching the theoretical composition from the oxides in the presence of carbon.

Table 2 gives grain-size measurements made under the microscope. The oxides gave much smaller grain sizes, as one would expect, for the carbon hinders the growth of the grains of nitride.

The powders were rendered convenient to handle by mixing them with a 5% solution of rubber in benzene (2 cc per 100 g); the mixture was dried for 1 hr at 80°, and cylinders 7.8 mm in diameter and 6 mm high were pressed in a steel die to give porosities not exceeding 30%. The mass of titanium nitride was 0.85 g and the pressure was 1 t/cm<sup>2</sup>; the corresponding figures for zirconium nitride are 1.2 g and 1.2 t/cm<sup>2</sup>.

Table 3

Sintering Conditions

Nitride of	Heating 1		Heating 2	
	°C	min	°C	min
Ti*	500-600	45	1400	60
Zr*	"	"	1450	60

\*res. pressure Hg col: 1-2·10<sup>-3</sup>

The specimens were heated in a vacuum oven having a glass bulb and a molybdenum heater to increase their strength and remove the bonding agent (Table 3). Table 4 gives the limits of variation of the porosity and density, as well as the compositions.

Table 4

Preparation of Briquettes from Nitrides

Property	Nitride		
	Ti	Zr	Ti, from oxide
Porosity, %			
pressed	23-38	39-42	42-45
sintered	31-34	38-39	37-39
S. g., g/cm <sup>3</sup>			
pressed	3.2-3.3	4.0-4.1	2.8-3.0
sintered	3.4-3.6	4.2-4.3	3.2-3.3
N, %			
before	21.6	12.97	20.20
after	20.77	12.45	19.02
Formula			
before	TiN	ZrN	TiN
after	TiN <sub>0.96</sub> TiN <sub>0.92</sub>	ZrN <sub>0.97</sub> ZrN <sub>0.93</sub>	TiN <sub>0.89</sub> TiN <sub>0.84</sub>

The proportion of nitrogen is less than theoretical, mainly as a result of the residual oxygen, though there may have been a tendency for Mo-Ni solid solutions to form as a result of decomposition of the nitride under vacuum. Both effects can affect

the wetting by molten metals; the first tends to hinder it and the second to facilitate it, for oxides are usually less readily wetted than clean metals.

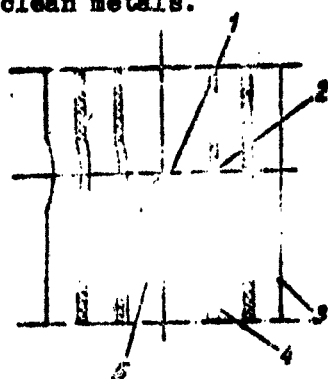


Fig. 1. Apparatus for impregnating briquettes: 1) drop of metal, 2) specimen, 3) molybdenum screen, 4) molybdenum heater, 5)  $\text{Al}_2\text{O}_3$  support.

The specimens were tested for wetting and impregnation with nickel and cobalt in the furnace with the molybdenum heater, the residual pressure being 1-2  $\mu\text{Hg}$ . A thin pressed disc of the powdered metal was placed on top of the specimen, which rested on an alumina plate. The temperature was measured (with an optical pyrometer) and the metal was observed via the glass bulb, there being holes in the heater and screen for this purpose (Fig. 1). This gave the temperature of the drop of metal to  $\pm 20^\circ\text{C}$ ; the correction for absorption in the glass was taken as  $50^\circ$ . The cobalt powder had the following composition (%):

99.5 Co, 0.22 Ni, 0.10 C, 0.01 Fe, 0.06 Si, and 0.07 water; the nickel: 99.56 Ni, 0.07 Co, 0.10 C, 0.01 Fe, 0.056 Si, 0.04 C, 0.016 C; 0.14 water. The specimens were brought up to temperature over a period of 45 min and were kept at temperature for 30 min; Table 5 gives the results, including the mean contact angles. Figure 2 shows the microstructure of impregnated titanium nitride.

Table 5

Impregnation Tests							
Co				Ni			
°C	Porosity, %		Angle, deg	°C	Porosity, %		Angle, deg
	before	after			before	after	
TiN from metal							
1490	32	7.9	28	1450	34	20	36.5
1560	31	8.4	-	1520	34	3.7	-
1640	33	4.4	-	1600	33	9.8	-
TiN from oxide							
1490	37	37	-	1450	37	37	-
1560	38	38	69.5	1520	37	37	47.5
1640	37	37	-	1600	37	37	-

Fig. 2. Sections of TiN briquettes impregnated with a) Ni, x 600; b) Ni, x 1500; c) Co, x 1500

			ZrN from metal			
1490	38	38	-	1450	38	38
1560	-	-	56	1520	39	39
1640	-	-	28	1600	39	39

[Available photograph  
not suitable for  
reproduction]

It was not always possible to measure the contact angle at the highest temperatures on account of pronounced bubbling of the metal. The values given are only approximate, on account of the small number of measurements and the porosity. However, the impregnation is reasonably complete, and the angles are less than  $90^\circ$ , so wetting does occur.

The metals give nitrides of more nearly stoichiometric composition, but the nitrogen content is still too low. Extended nitriding gives no improvement, and sometimes even the reverse, probably because some of the nitrogen is replaced by oxygen that leaks into the furnace. The loss of nitrogen is especially prominent during vacuum sintering, because a solid solution is formed. The incomplete wetting is associated with the residual oxygen, for oxides are less readily wetted. Adsorbed gases also prevent the molten metal from penetrating into all the pores.

The cobalt began to melt at  $1490 \pm 20^\circ$ , and the nickel at  $1450 \pm 20^\circ$ ; melting always started at the contact with the specimen [5]. The metal usually began to wet the sample somewhat after it had melted completely; the contact angle fell gradually

from  $90^\circ$  to the value given in Table 5, thereafter remaining constant. The size of the drop decreased when the metal impregnated the sample, but the shape remained the same.

Complete impregnation was attained only for titanium nitride made from the metal; the finely divided carbon and residual oxide hindered the impregnation of materials derived from oxides, and the very small grains also tended to interrupt the capillary channels.

The impregnation was hindered by external factors, not by lack of wetting, for a thin film of metal remained adhering to the surface of the specimen after cooling; this film was formed by the flowing away of the metal, and it would not be present if the material were not wetted.

The impregnated titanium nitride contained two very different phases, namely rounded and prominent grains of nitride (in places intergrown with loss of boundaries) and an extensive uniform network of metal between the grains. The pseudobinary combination of nitride with cobalt (or nickel) may be of eutectic type, as a result of limited solubility of the nitride in the metal. The reduction in the melting point of the metal at the contact with the grains also indicates that the product is a eutectic. The solubility of the nitride in the metal is also confirmed indirectly by the very much enlarged grains of nitride (relative to the initial grain size); this points to recrystallization via the liquid state, which would be impossible unless the nitride dissolves in the liquid but has a limited solubility in the solid state. The ratio of the volumes of the two phases as seen on the photomicrographs corresponds roughly with the residual porosity as calculated from the volume and weight of the impregnated specimen.

### Conclusions

1. Titanium nitride is wetted by cobalt and nickel; the contact angles are much less than  $90^\circ$ .
2. Porous briquettes pressed from titanium nitride can be impregnated by molten metals; the purity and grain size are the controlling factors.
3. Titanium nitride made from the high-purity metal can be impregnated reasonably completely by cobalt and nickel; the oxide gives a product that is wetted but not impregnated, which is attributed to contamination with free carbon and to the small grain size.
4. Zirconium nitride made from the metal is wetted by cobalt and nickel, but it was not impregnated under the conditions used. The reason is considered to be as above; some residual oxygen may also have an effect.

5. Some suppositions are made as to pseudobinary combinations of titanium nitride with nickel and cobalt, which appear to be eutectic, the nitride being of limited solubility in the metal in the solid and liquid states.

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CSO: 1879-N

## CAUSES OF LAMINATION CRACKS IN PRESSINGS MADE OF METAL POWDERS

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Moscow, *Izvestiya Vysshikh Uchebnykh Zavedeniy, Chernaya Metallurgiya*  
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The formation of these cracks is usually represented as follows. Expansion in the section a-a (Fig. 1) during ejection causes cracks to appear [1]; the extent of the elastic aftereffect at right angles to the pressing direction is 0.15-0.30% for the best types of industrial powders and may be as much as 1% for powders of poor quality [1]. The elastic stretching of the cylindrical steel die is usually neglected, but ones having ratios of outside to inside diameters of 1.4-3.0 give [2] expansions of 0.039 to 0.676% in response to pressures of 500 to 4000 kg/cm<sup>2</sup>, so the effect cannot be neglected. The following observations illustrate this.

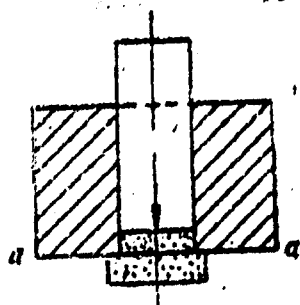


Fig. 1. Formation of cracks [1].

1. Cylinders 10.25-10.65 mm high were pressed from grade AM iron powder at a fixed pressure of 4 t/cm<sup>2</sup> [3], the dies having an internal diameter of 25.05 mm and an external one of 40 to 100 mm. All specimens from the 40 mm die showed cracks (Fig. 2).

2. Brush inserts of trolleybuses have been observed to have laminated cracks after pressing; similar cracks are found when specimens 5 mm high were pressed in the same die (internal hole 60 mm long, 20 mm wide)\*. These cracks occur along the specimens on the sides (Fig. 3) at the point where the die was

\* Pressings made by M. N. Lotovich.

maximally deformed. All cracks were curved. The elastic af-

[Available photograph not suitable  
for reproduction]

Fig. 2. Layered cracks in cylinders of iron powder.

tereffect cannot explain cracks of this shape and position.

3. Two dies were made of grey cast iron; one of these was enclosed in a shroud made of St. 3 steel in the hot state.

These dies were used with AM powders to press cylinders of various heights at a fixed pressure of 4 t/cm<sup>2</sup>. All specimens from the unsupported die showed cracks (Fig. 4a), but only the highest ones from the supported die (Fig. 4b).

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reproduction]

These observations show that elastic stretching of the die is one of the main causes of cracking; the die expands during the pressing but contracts when the pressure is released. This contraction is opposed by the friction between specimen and die, and also by the resistance of the specimen.

Fig. 3. Layered cracks on a rectangular specimen.

The elastic deformation may vanish as soon as the pressure is removed, before the specimen is ejected, or during the ejection; the specimen simultaneously expands (elastic aftereffect). The condition for the specimen to be free from cracks may be put as

$$\epsilon d / \sigma + \gamma \approx \delta$$

in which  $\delta$  is the elastic deformation of the plunger,  $d$  is the diameter or other dimension of the die perpendicular to the pressing direction,  $s$  is the total wall thickness of the specimen in the direction of  $d$ ,  $\nu$  is the relative deformation of the specimen as a result of elastic expansion, and  $\delta$  is the permissible deformation of the specimen. This  $\delta$  is dependent on the mode of deformation as well as on the properties of the material; the  $\delta$  for hydrostatic compression is larger than that for compression in a plane or along a line. The more nearly the stress distribution is to linear, the smaller  $\delta$  [4].

[Available photograph not  
suitable for reproduction]

Fig. 4. Specimens of iron powder pressed in cast-iron dies: a) without shroud; b) with shroud.

The above explanation of the cracks appears to agree closely with practical observations. Elastic stretching of the die and the elastic aftereffect in the specimen are the main causes of cracking. Stronger dies provide an effective means of minimizing cracking in powdered-metal specimens, for they show less elastic deformation.

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# PRESSING OF METAL POWDERS

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Moscow, Vestnik Mashinostroyeniya (Herald of Machine Building)  
No 5, May 1959, pp 53-56

Bal'shin [1], Unökel [2], and others have made fundamental studies on the pressing of metal powders, but some aspects of considerable importance in practical powder metallurgy have been neglected. These include the effects

Table 1

Time, min	Pressure, t/cm <sup>2</sup>					
	2		4		6	
	Briquettes from					
	p*	s	p	s	p	s
	Iron					
0	4.30	4.10	5.0	4.90	5.65	5.32
0.5	4.35	4.06	5.08	4.92	5.75	5.63
1.5	4.37	4.06	5.25	5.10	6.00	5.68
3	4.4	5.25	5.39	4.88	6.74	5.83
	Tungsten					
0	8.55	9.95	10.40	11.00	11.00	11.80
0.5	8.70	9.80	10.60	11.20	11.21	11.80
1.5	8.84	9.95	10.25	11.50	11.50	12.40
3	9.03	9.76	11.31	10.40	11.52	14.20

\*p = pressing, s = sintering.

4.2  $\mu$  was also used. The powders were pressed in a cylindrical steel die at 2, 4, or 6 t/cm<sup>2</sup>, the density being measured as a

of the time under pressure on the density, elastic after-effects, effects of repeated pressing, and the distribution of the forces during pressing.

Pressing  
Time. Iron powder (poured density 2.06 g/cm<sup>3</sup>) was prepared by reduction from rolling scale as was sieved (025B sieve); tungsten powder (3.28 g/cm<sup>3</sup>) of mean grain size

function of pressing time (times of 0.5, 1, 1.5, and 3 min were used). The iron samples were sintered in hydrogen at 1000-1150° and those of tungsten at 2100°, in both cases for 1 hr. Table 1 gives the densities in g/cm<sup>3</sup>.

The density of an iron specimen generally increases with the pressing time, the increase itself tending to rise with the pressure. This is a result of liquidation of the state of stress (distribution of the stress throughout the volume). The loss of density on sintering indicates that the internal stresses are thereby relieved. On the other hand, the tungsten specimens became denser on sintering, which may mean that the forces resulting from sintering substantially exceed those of the stresses, which is associated with the high volatility and ready reduction of tungsten oxides, so sintering is activated.

Elastic Aftereffect. Here the dimensions of the specimens were measured under pressure and after pressing (a steel die 12.3 mm in diameter was used) for tungsten, tungsten carbide, iron, copper, and aluminium. Each powder was used in three different grain sizes (Table 2). Figure 1 shows the results.

Table 2

Powder	Sep. method	Fraction, $\mu$		
		Large	Med.	Small
W	Sedim.	1.6	1.3	0.7
WC	"	2.0	1.5	0.7
Cu	Sieve	75-85	42-75	42
Fe	"	105-150	42-85	42
Al	"	10-30	85-105	42

The large and medium fractions showed less effect; the maximum effect with WC moved to lower pressures as the grain size was reduced, which is evidently a result of brittle fracture of the particles. There is a fairly pronounced trend in the effect with Fe, Cu, and Al; there is also an increase with the hardness of the powder, as has been observed previously /1/, but this extends only up to a certain pressure,

past which point there is a fall, which is caused by brittle fracture and hardening in the grains. The effect also varies with time; for example, copper specimens left in a non-oxidizing atmosphere for a week after pressing increased in diameter by 0.14-0.36% and in length by 0.36-0.48%.

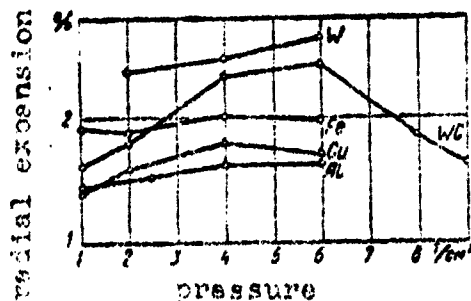


Fig. 1. Relation of elastic aftereffect to pressure.

Effects of Repeated Pressing. The powders

detailed in Table 3 were used here in a steel die 12 mm in diameter; after pressing, the specimens were ground up, sieved, and pressed again. Table 4 gives the results. The density of copper powder in all three forms increased steadily from

Table 3

Grain size, $\mu$	% content	
	Cu	W
42	-	1
42-25	-	1.5
25-18	-	2.65
18-15	-	17.35
150-105	0.15	18.9
105-85	1.3	34.1
85-75	1.5	8.25
75	97.05	16.15

Note. Poured and tamped densities 1.63 and 2.33 (Cu), and 4.52 and 6.74 (W).

pressing I to pressing III, provided that the pressure used in pressing III was equal to or greater than that used in I and II; but if III was (say) 4 t/cm<sup>2</sup> whereas I and II were 8 t/cm<sup>2</sup>, the reverse occurred. The effects of repeated treatment on the much harder metal tungsten were very much less, though the trend in the density of the pressed specimens was as for iron powder (pronounced fall if pressure III was below pressures I and II). This method enables one to produce the most favorable distribution of particle sizes and can be used to increase the density of the pressed materials.

#### Force Distribution. Unckel

[27] has used the die shown in Fig. 2 to measure the forces in pressing;

between the guide and the lower support there is a ring of copper or soft iron, which is indented by four steel balls held in recesses. A similar disc supported by three balls

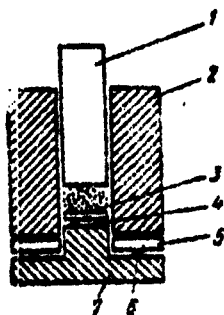


Fig. 2. Unckel's die for determining the stress distribution in pressing: 1) plunger; 2) guide; 3) support for powder; 4) balls; 5) ring support under guide; 6) steel balls; 7) base of die.

records the force acting directly on the powder; the difference represents the frictional force  $N$  between the powder and the wall of the die. The indents are used to deduce the forces as follows:  $N$  is given by

$$N = \mu P_w F_w,$$

in which  $\mu$  is the coefficient of friction,  $P_w$  is the lateral pressure, and  $F_w$  is the area of contact with the wall. Now

Effects of pressings I, II, and III at pressures  $P$  ( $t/cm^2$ ).22

$P_w$  is given [3] by

$$P_w = \frac{2}{3}P,$$

in which  $\frac{2}{3} = \nu/(1 - \nu)$  and  $\nu$  is Poisson's ratio,  $P$  being the pressure applied to the plunger;  $\frac{2}{3}$  is not affected by the porosity. Then we have that  $N/N_p = 4\frac{2}{3}\mu H/D$  for the proportion of the pressing force  $N_p$  lost by friction. This increases with  $H$ , and so decreases as  $N_p$  is increased, as Unckel has shown, his values for the ratio for iron powder being 70% at 2 t/cm<sup>2</sup>, 60% at 4, 50% at 12, and 34% at 18. A lubricant mixed with the powder reduces the ratio (to 41% at 12 and 18 t/cm<sup>2</sup> in Unckel's case).

We have used the same method, but with special attention to lubrication of the walls as a means of reducing the friction. The powders were of Cu, Fe, and W, which are metals that differ greatly in plasticity. The Cu powder was made by reducing copper oxide with hydrogen (grain size 2-3  $\mu$ , poured density 1.46 g/cm<sup>3</sup>); the Fe, by reduction of scale with hydrogen (2-3  $\mu$ , 2.0 g/cm<sup>3</sup>); and the W, by reduction of tungstic anhydride (5-6  $\mu$  4.22 g/cm<sup>3</sup>). The lubricant was a 4% solution of paraffin wax in benzine (150 cc per 100 g of powder). The powders were pressed without lubricant, with the wall and ends lubricated, and with lubricant incorporated in the powder; the pressures ranged from 0.4 to 2.5 t/cm<sup>2</sup>. Table 5 gives the results, which indicate a type of variation rather different from that reported by Unckel; the reason is that he used much higher pressures.

The proportion of the force lost by friction tends to increase with the pressure if the die is unlubricated, but it decreases if die or powder contains lubricant. The first effect may be caused by seizure and the second by leakage, which tends to increase with pressure. Unckel did not observe seizure, for the pressures he used were too high for the force to be important. Iron powder here gives a pronounced effect, which is greater than that for the Cu-Fe pair; the lubricant here decreases the proportion lost but does not affect the trend of the proportion with pressure.

The curve for  $P_1 f(P)/(P_1 + P_2)$ , in which  $P$  is the pressure applied, has a maximum for tungsten powder when the wall is lubricated but decreases as  $P$  increases if the powder contains lubricant, as for iron. The reason is that low pressures are insufficient to overcome the seizure of the powder with the wall of the die, especially when there is no lubricant. A lubricant within the powder tends to suppress seizure altogether, because the lubricant tends to be extruded along the wall as the pressure increases.

Table 5

Lub. method	$P_1$ , t/cm <sup>2</sup>	kg		$P_1/(P_1 + P_2)$ as %	Res. por., %	$H_B$ , kg/mm <sup>2</sup>	$\sigma$ , kg/mm <sup>2</sup>
		$P_1^*$	$P_2$				
Cu							
None	0.7	616	776	44.0	49.1	-	2.94
"	1.2	1275	1180	52.0	39.2	17.0	5.54
"	1.6	1645	1630	50.0	29.2	33.5	9.60
"	2.0	2240	1665	54.0	25.0	42.0	12.6
Wall of die	0.7	470	1028	34.0	48.6	-	2.59
"	1.1	585	1282	27.0	36.8	16.6	3.50
"	1.5	755	2265	23.0	23.1	31.2	7.00
"	2.1	960	2840	23.0	17.2	39.3	10.56
In powd.	0.7	468	812	35.0	47.3	-	2.87
"	1.0	610	1312	31.0	39.4	15.8	4.70
"	1.5	735	2240	24.0	24.4	29.6	8.04
"	2.0	894	3100	22.0	15.6	38.7	10.9
Fe							
None	0.4	412	470	46.8	52.7	-	3.02
"	1.1	1145	978	54.4	45.5	25.2	4.01
"	1.6	1763	1451	55.0	41.1	38.2	5.75
"	1.9	2257	1510	59.9	33.8	46.3	9.34
"	2.3	3290	1354	71.1	31.7	50.1	9.9
Wall of die	0.4	207	538	27.8	51.7	-	0.59
"	0.9	518	1367	27.5	41.3	21.2	4.07
"	1.4	1140	1696	40.4	35.2	30.3	4.4
"	1.7	1477	1990	42.6	32.9	46.3	8.7
"	2.1	2021	2234	47.5	25.9	71.5	11.8
In powd.	0.5	466	513	47.6	50.1	-	-
"	1.1	915	1309	41.5	41.4	18.1	4.8
"	1.4	1319	1524	46.2	36.2	26.7	5.1
"	1.6	1624	1456	52.2	31.8	34.6	9.6

\* $P_1$  = force on outer,  $P_2$  = force on powder.

(Cont. over)

Table 5 (Cont.)

Lub. method	P, t/cm <sup>2</sup>	kg		P <sub>1</sub> /(P <sub>1</sub> + P <sub>2</sub> ) as %	Res. por., %	H <sub>B</sub> , kg/mm <sup>2</sup>	σ, kg/mm <sup>2</sup>
		P <sub>1</sub>	P <sub>2</sub>				
W							
None	0.5	1110	723	58.2	47.1	-	0.83
"	1.1	997	1195	45.5	46.9	-	0.83
"	1.9	1870	1940	49.5	46.9	-	0.83
"	2.5	3010	1990	60.0	40.0	-	2.34
Wall of die	0.5	495	565	46.5	48.0	-	0.28
"	1.1	975	1178	44.5	47.0	-	0.28
"	1.4	1335	1550	40.5	46.8	-	0.28
"	1.6	1610	1710	48.5	43.0	-	0.38
In powd.	0.6	447	660	39.4	51.0	-	0.28
"	1.2	1010	1340	43.0	49.5	-	0.38
"	1.7	1280	2120	37.7	47.5	-	0.48
"	1.9	1612	2240	47.0	44.0	-	0.51
"	2.3	2500	2142	53.6	38.6	-	0.63

The residual porosity falls steadily as P increases, the fall being most rapid for copper, as one would expect (by about a factor 2 for unlubricated powder and by a factor 3 for lubricated). The effect is less for iron (factors of 1.5 and 2 respectively) and least for tungsten (1.2 and 1.5).

The hardness and yield point σ in compression both increase with P; the lubricant reduces σ for Cu and W but has little effect for Fe, which may be a result of differences in the interaction between the lubricant and the powder (the lubricant is active with respect to iron but inert for Cu and W, where its presence enables the particles to slide over one another). The low strength of tungsten specimens occurs because the pressures were too low for use with this hard material.

The conclusions to be drawn are as follows.

The density increases with the pressing time; the internal stresses are thereby reduced. The elastic aftereffect in

the more plastic metals generally decreases as the grain size increases; with brittle materials of carbide type, it increases only up to pressures insufficient to cause brittle fracture.

Repeated pressing and powdering provides a substantial increase in the density of the pressed products without any great increases in the internal stress and elastic aftereffect.

The distribution of forces in the pressing of powders is very much dependent on the seizure between the die and the powder, especially for low pressures and low rates of pressing.

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